

NAVAL POSTGRADUATE SCHOOL

Monterey, California



AMMONIUM PERCHLORATE AND AMMONIUM
PERCHLORATE - BINDER SANDWICH COMBUSTION

by

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ABSTRACT:

Single crystal and pressed polycrystalline ammonium perchlorate (AP) wafers, pressed polycrystalline AP pellets, and ammonium perchlorate-binder (polybutadiene acrylic acid, hydroxyl terminated polybutadiene, polyurethane, and carboxyl terminated polybutadiene) sandwiches were burned in a schlieren equipped combustion bomb and in a centrifuge mounted combustion bomb. High-speed color schlieren and regular motion pictures were taken of the combustion process to determine the effects of pressure, binder type, binder thickness, and purity of ammonium perchlorate on the AP deflagration. AP pellet fabrication techniques and burning rate acceleration sensitivity were also investigated.

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I. INTRODUCTION

Many analytical and experimental studies have been made of composite solid propellants which use ammonium perchlorate (AP) as the oxidizer. These have been reviewed by numerous investigators, e.g., Varney (12). In addition, considerable effort has been made to understand AP deflagration and AP-binder sandwich combustion. Two-dimensional propellant sandwiches have been used in order to provide a convenient means of studying a variety of propellant types under a wide range of test conditions, and they allow AP-binder interactions to be conveniently studied by visual methods.

While much has been learned about AP and AP-binder sandwich decomposition and deflagration, many questions remain unanswered. Two such questions are concerned with the effects of pressure and binder composition on flame type (premixed or diffusion), and flame characteristics (laminar, turbulent, steady, or nonsteady). If adequate modeling of propellant combustion is to be obtained, the answers to these questions are required.

In addition, much speculation still exists concerning the effects of impurities and strand construction technique (molding pressure, etc.) on AP deflagration.

In the past, most studies of AP-binder sandwich combustion and AP deflagration have been conducted by using high-speed motion picture photography and post-fire examination of quenched samples (1-9,15). In order to gain further needed knowledge of the behavior of AP during deflagration, Kennedy (10) conducted a schlieren investigation of AP-binder sandwich combustion. He found (a) a complex interaction occurs between the primary flame, AP deflagration, and binder pyrolysis products, (b) the sandwich burner flames appeared to be laminar below the lower pressure deflagration limit (PDL) of AP and unsteady or "turbulent" above the PDL of AP, and (c) two distinct multiple flame regions

exist, one above the binder and one near the binder-AP interface. Limitations of his initial study were (a) the use of a schlieren optical depth that allowed much flame averaging to occur, (b) a magnification on the film plane of 0.33, which did not allow sufficient resolution of the burning process, (c) separate schlieren and real-light films of different propellant burners, burning under similar conditions, and (d) the use of only one grade of AP and of only one type and thickness of binder.

In this investigation, some of the refinements made to the apparatus and the techniques used by Kennedy were (a) a higher object magnification on the film plane, (b) color schlieren and real-light photography of a single sandwich burner on the same film, and (c) a smaller sandwich to reduce the schlieren optical path length through the flame, thereby reducing the averaging effect on the schlieren photography.

In previous studies of AP deflagration (11,14) it has been reported that the burning rate of polycrystalline AP pellets is dependent upon the initial size of the crystals employed. There has also been much discussion in the literature concerning the effects of impurities on AP deflagration. However, most of these studies did not deal with impurities normally found in commercial grade AP. In a study of the burning rate acceleration sensitivity of nonmetalized composite propellants, Netzer (10) investigated the effects of impurities and pellet fabrication technique upon the measured burning rate of AP. Major results of that study were (a) regression rate is not affected by initial crystal size used in polycrystalline AP pellets if high molding pressures are used, (b) impurities normally found in commercial grade AP (sulfated ash, etc.) inhibit the regression rate, (c) an AP melt exists on deflagrating AP in the pressure range from 400 to 800 psi. The quantity of melt that remains on the surface is

a function of ignition energy and the surface configuration, and (d) acceleration directed normal and into the burning surface acts upon the liquid melt, decreasing the regression rate with increasing acceleration.

Limitations of the above study were (a) only one pressure was used (500 psi) to examine the effect of acceleration on burning rate, (b) the pressure range investigated at standard gravity conditions was limited to 800 psi for pure AP, and (c) only one pressure (500 psi) was used to study commercial grades of AP.

The purposes of this investigation were (a) to refine the experimental apparatus and the techniques used by Kennedy (10) in order to improve the quality of the experimental data, (b) to use the improved methods to study AP deflagration and AP-binder sandwich combustion for various purities of AP and types and thicknesses of binder, and (c) to extend the AP pellet deflagration work of Netzer (10) to include a wider range of operating pressures and accelerations and additional pellet fabrication methods.

II. METHOD OF INVESTIGATION

The investigation consisted of three related studies: (a) a study of polycrystalline AP pellet deflagration, (b) a schlieren study of the deflagration of pressed polycrystalline AP wafers and single crystals of pure AP, and (c) a schlieren study of AP-binder sandwich combustion.

Propellant sandwiches were made from three grades of pressed polycrystalline AP and three different types of binder. The same three grades of AP were also used in pellet form. The grades of AP were ultra-high purity (UHP), commercial grade, and commercial grade with tricalcium phosphate (TCP) added as an anti-caking agent. The three binders used were polybutadiene acrylic acid (PBAA), polyurethane (PU), and hydroxyl terminated polybutadiene (HTPB). The binder thicknesses were varied from 25 microns to 508 microns for sandwiches of PBAA. For sandwiches of PU, a binder thickness of 51 microns was used. For sandwiches of HTPB, the binder thicknesses were varied from 25 microns to 51 microns. Sandwiches made from single crystals of AP and carboxyl terminated polybutadiene (CTPB) were also studied.

The two schlieren studies were conducted in a nitrogen purged combustion bomb at pressures from 100 to 1000 psig. A high-speed (7500 FPS) color motion picture was taken for each test condition. Each film contained alternating frames of color schlieren and standard real-light color pictures. The films were analyzed to determine the effects of binder type, binder thicknesses, AP type and purity, and combustion pressure on the visible flame, the AP deflagration and burning surface characteristics.

Polycrystalline AP pellets of various purity were burned in standard and high acceleration environments. Pressures from the low pressure deflagration limit to 1250 psi and accelerations from zero to 1000g were employed. These

tests were conducted to determine the acceleration sensitive mechanisms in AP deflagration. In addition the effects of impurities and pellet molding pressure and time on the burning rate were investigated.

III. EXPERIMENTAL APPARATUS AND PROCEDURES

A. Propellant Specifications

Specifications for the ammonium perchlorate utilized in this investigation are shown in Table I. In addition, single crystals of pure AP were provided by the Naval Weapons Center.

The composition and cure data for the binders used in the fabrication of the sandwich burners are presented in Table II. Sandwiches made from single crystals of pure AP and CTPB binder were also supplied by the Naval Weapons Center.

B. High Acceleration Centrifuge

The centrifuge has been discussed in the literature (10). Polycrystalline AP pellets were rigidly inhibited on all but the burning surface. Ignition was accomplished using a black powder/glue/acetone mixture on the AP surface with an adjacent resistance wire. A few tests were conducted using only a nichrome resistance wire placed directly on the AP surface.

Tests were conducted to $\pm 1000g$ (+, normal and into and -, normal and out from the burning surface).

Burning rates were determined from the known pellet thicknesses and the burn times obtained from the pressure-time traces. Post-fire residue in the rigid inhibitors was also examined after each test.

C. Combustion Bomb and Schlieren

Details of the combustion bomb are discussed in Reference 10. Previous work done at the Naval Postgraduate School (10) with the combustion bomb used a 203 millimeter lens to focus the image of the deflagrating sandwich burner onto the film plane of the high-speed motion picture camera. This lens did not provide the desired resolution of the deflagrating AP and sandwiches. Consequently,

a 610-millimeter lens was substituted and a magnification of 0.8 was achieved on the film plane.

The alignment of the schlieren light source, the various lenses, the combustion bomb, and the camera was critical. To facilitate the alignment procedure, an Optics-Technology He-Ne laser was used to ensure that the lenses, bomb, and light source were in as straight a line as possible. Figure 1 is a schematic of the combustion bomb and schlieren system used in this investigation.

Combustion bomb pressurization, burner ignition, and camera activation were controlled from behind a safety shield [See Fig. 2].

Ignition of the sandwiches was accomplished using a nichrome resistance wire placed across the top of the sandwich.

In earlier studies a color matrix constructed by placing a red and a blue gelatin filter between two plates of glass was used to produce the color schlieren. To improve the schlieren quality, a color slide photograph of the red and blue gelatin filters between two plates of glass was used in this study.

In order to make high-speed motion pictures in which both schlieren and real-light photography of a single burner could be made, a light-source chopper was designed and constructed. At full camera speed the chopper provided approximately four frames of real-light photography alternated with four frames of schlieren photography.

A model K2004E-115 Hycam camera with variable framing rates from 1000 to 10,000 pictures per second and a pulse timer were used to take the schlieren and real-light photography. Kodak Ektachrome 7241, ASA 40, high-speed color film was used for all photography.

The light source for the real-light photography was a modified Spindler and Sapppe Inc., Selectroslide, model SIM-1200 (1200 watt) slide projector aimed through the 45° window of the combustion bomb. The light source for the schlieren photography was a 1000 watt mercury arc lamp.

D. AP Pellet, AP Wafer, and Sandwich Fabrication

AP was pressed into pellets and wafers using compaction molds similar to that used by Varney (12). Fabrication techniques have been discussed in Reference 10.

Prepared sandwich burners were measured for height, width, binder thickness, and optical depth [See Fig. 3] with a Gaertner Scientific Corporation microscope.

IV. RESULTS AND DISCUSSION

A. Pressed AP Pellet Deflagration in Standard and High Acceleration Environments

This investigation extended the earlier work reported in Reference 10. To facilitate a complete discussion most of the results obtained from Reference 10 have been combined with the results obtained in this investigation.

Before the acceleration sensitivity of AP deflagration could be studied it was first necessary to consider the effects of pellet fabrication techniques on the burning rate and general combustion characteristics.

Previous work (10) had indicated that the rigid inhibitor had negligible affect upon the burning rate. In addition, it was found that compaction times greater than 20 minutes (up to 24 hours) had no appreciable affect upon the burning rate.

A series of tests were conducted to determine the effects of short compaction molding time and compaction pressure. The results are presented in Figure 4.

From Figure 4 it is observed that compaction time is more important than compaction pressure for obtaining high density pellets that exhibit burning rates that are in agreement with previously published data (also see Figure 6). Based upon these results, all subsequent pellets (and wafers) were fabricated using a compaction pressure of 30,500 psi and a compaction time of 20 minutes. Scanning electron microscope (NWC-China Lake) photomicrographs also indicated that this fabrication technique provided pellets, which had very few internal voids (see Reference 10).

Another series of tests were conducted to determine the effect of AP crystal size on the pellet burning rates. These tests were conducted because other studies (11,14) had indicated that increasing AP crystal size decreased pellet

burning rate. The results are presented in Figure 5 and indicate that initial crystal size had no affect upon the pellet burning rate. Based upon these results, all subsequent pellets (and wafers) were made using the as-received ammonium perchlorate.

Impurities in AP significantly affect the burning rate (13). However, the impurities normally found in propellant grade AP have not received adequate attention. An investigation was conducted to determine the affect of impurities on the AP burning rate. The results are presented in Figure 6. The burning rates of the ultra-high purity pellets are in good agreement with single crystal data. Sulfated ash and/or tri-calcium phosphate inhibit AP regression rate and may increase the effective low pressure deflagration limit. An interesting question arises with regard to AP deflagration in a composite propellant. If propellant grade AP with TCP conditioner is consumed in a composite propellant in the same manner as it is in pellet form, and if the existence of an AP melt is associated with operation above the low pressure deflagration limit (2,4), then AP melt may not exist during combustion of propellants which use this grade of AP.

Figure 7 shows the affect of acceleration on the burning rate of ultra high purity AP pellets. The first thing to note is the data scatter obtained in acceleration environments. This is in contrast with the highly reproducible burning rates measured under standard gravity conditions (see Figure 6). This variation in burning rate is also typical of nonmetallized composite propellants which are burned in acceleration environments and indicates that AP surface melt is the probable source of the varied behavior.

At 500 psi and 1000 psi, no burning was accomplished above 250g and 500g respectively. Previous work (10) had shown that a surface melt of AP existed on these "no-burn" tests.

When the black-powder/glue ignition system was replaced by a single ignition wire across the center of the pellet, burning was successful to 500g at 500 psi.

However, the burning rate remained less than the zero-g rate with black-powder/glue ignition ($r/r_0 = .875$). With the acceleration directed out from the burning surface the burning rate remained practically unchanged from the zero-g rate.

The burning rate acceleration sensitivity of AP is apparently a surface melt phenomena. The quantity and location of surface melt are a strong function of pressure (4). At 1000 psi AP melt is restricted to valleys in a fine ridge-valley structure (4). This fact is the most probable explanation for the results shown in Figure 7, i.e., the tests conducted at 1000 psi (smaller quantities of localized melt) would burn to a higher acceleration level than tests conducted at 500 psi (continuous bubbling froth).

The burning rate acceleration sensitivity of AP at both 500 and 1000 psi are opposite to that observed for nonmetallized composite propellants. Thus, AP/AP melt interactions with binder/binder melt are more plausible mechanisms for the burning rate acceleration sensitivity of nonmetallized composite propellants.

B. Schlieren Study of AP and AP-Binder Sandwich Combustion

B.1 Data Summary

Table III presents a summary of the tests conducted in this investigation. Table IV summarizes the major experimental apparatus employed in the study.

Because of the cost of color reproductions, only black and white prints (with identifying marks) have been included in this report. A color reproduction of Figure 8 through 13 is available on loan from the authors. In addition, an edited color film has been prepared which includes segments of all AP and AP-binder sandwich films.

One film was taken of a very wide wafer of pressed polycrystalline AP, burned in order to determine if the flow of nitrogen in the combustion bomb had any effect on the temperature profile as shown by the schlieren photography. There was no noticeable effect.

Two runs with HTPB sandwiches of pressed polycrystalline AP with TCP added were attempted at pressures of 300 psig and 500 psig. Neither achieved successful ignition at these pressures and therefore were not included in the tabulated data.

B.2 Ammonium Perchlorate Deflagration

The lower pressure deflagration limits (PDL) for the various grades of AP burning as a monopropellant were determined prior to any data points being run. Ultra-high purity (UHP) and commercial grade AP had the same PDL, approximately 355 psig. This is a contrast to the larger AP pellet data where the PDL of pure AP was somewhat lower than the PDL of commercial grade AP. All attempts to ignite AP with TCP in the pressure range from 100 psig to 1000 psig were unsuccessful.

The color schlieren taken at 400 psig of pressed polycrystalline ultra-high purity (PP-UHP) AP, single crystal ultra-high purity (SC-UHP) AP, and pressed polycrystalline commercial grade (PP-COMM) AP all had blue and red color shifts just above the burner surface. Figure 8 shows a typical color schlieren, taken of a single crystal at 400 psig. These color shifts are on the order of 300 microns in width. The deflagration of all three burners appeared laminar, with a definite temperature peak above the center of the burner. The PP-COMM AP had a scalloped surface which accounted for a more unsteady appearance of the gas flow above the deflagrating AP. In Figure 8 a yellow-green zone is visible, extending from the burner surface to a depth of approximately 200 microns. This zone is thought to be related to the phase change from an orthorhombic crystalline structure to a cubic structure (the 243°C isotherm) as reported by Boggs and Kraeutle (4), Hightower and Price (2), and Beckstead and Hightower (1). Beckstead and Hightower (1) found this phase change zone to be approximately 22 microns deep in single crystals of AP burned

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at pressures of 400 psi. The three studies mentioned above investigated quenched samples to determine the phase change zone thickness. The difference between their results and the results of this study may be due to removal of surface material during quench and/or to an inaccurate measurement of the penetration depth of the cubic structure obtained from the recrystallized quenched samples. More likely, however, is the possibility of light scattering (due to small cracks, etc.) and/or other than phase change phenomena causing the larger thickness found in this investigation.

Color schlieren taken at 500 psig of PP-UHP AP, SC-UHP AP, and PP-COMM AP indicated that the gases above the deflagrating AP were more turbulent in appearance than at 400 psig. There was much more large scale mixing closer to the burner surface than at 400 psig. Figure 9 shows a color schlieren photograph taken of a single crystal at 500 psig. The characteristic alternating red and blue zones were again visible and were on the order of 300 to 400 microns in width. The subsurface zone in Figure 9 appeared to be thinner than the zone in Figure 8, but was not well enough defined for accurate measurement.

Color schlieren taken at 800 psig of PP-UHP AP, and SC-UHP AP (see Figure 10) showed that the deflagration was turbulent, with a more uniform temperature in the gases above the burner. The subsurface zone was very thin at 800 psig.

Color schlieren taken at 1000 psig of a SC-UHP AP burner showed that the deflagration was extremely turbulent as evidenced by an almost uniform color in the gases just above the burner surface. The subsurface zone was not visible at this pressure. Careful examination of the film revealed the presence of alternating red and blue shifts very close to the surface. These color shifts were estimated to be on the order of 150 microns in width but were very difficult to measure because of their small size.

Comparison of the color schlieren taken of the single crystals indicated that the thickness of the subsurface zone associated with the crystal structure change (and/or other phenomena) decreased as pressure was increased in agreement with the results reported by Beckstead and Hightower (1).

The discussion of laminar vs. turbulent deflagration is somewhat debatable. At the intermediate pressures (500 - 800 psi), the gases are turbulent at some distance above the surface. However, close to the burner surface there may be a laminar region, i.e., there may be a "height to turbulence" as found in free-jet flames. At 1000 psi, the deflagration appeared turbulent very close to the AP surface.

Two additional tests were made in order to further study the subsurface zone. A color schlieren was taken of SC-UHP AP burned at 500 psi in which the color matrix was horizontally positioned. The schlieren thus indicated density (temperature) gradients only in the direction normal to the burning surface. The same subsurface yellow-green colored zone was evident, indicating that the green color resulted from the back lighting through the altered crystal structure (the mercury light source has several strong lines in the yellow-green frequency range) and was not due to schlieren effects.

Another interesting observation was made from the above film. The AP appeared to be burning in a pulsating, thermal layer manner. The red schlieren color above the crystal (which indicated an increasing temperature) evolved uniformly like puffs of smoke from the entire surface.

One additional test was made to investigate the subsurface zone. A single pure crystal of AP was wrapped with a resistance wire and the wire heated to approximately 250°C with a bomb pressure of 200 psi (below the PDL of AP). At each point where the wire touched the crystal the yellow-green color was evident.

These results indicated that the yellow-green subsurface region was definitely associated with the orthorhombic to cubic phase change.

The blue to red alternating color shifts across the burner surface mentioned above are thought to result from local zones of rapid density (or temperature) change, i.e., local reaction sites on the surface of the AP. Such local reaction sites have been reported by Boggs and Kraeutle (4) and Hightower and Price (2).

Hightower and Price (2) report surface depressions on the order of 150 microns in width on the burner surface of quenched samples burned in the range of 300 to 700 psi. The width of the blue to red color shifts measured from schlieren photographs taken in this study were about 300 microns in width. These blue to red color shifts may either be local reaction sites that merge together or may be indicating the spaces between the reaction sites.

The distance between reaction site centers was relatively independent of pressure for pressures between 400 and 800 psig. However, there was a distinct change in the appearance of the gases above the deflagrating surface when the pressure was raised from 800 to 1000 psig. The distinct sites which were evident at 800 psig became quite small or nonexistent at 1000 psig. Boggs and Kraeutle (4) found from quenched AP that the surface structure changed between 800 and 1000 psi. Below 800 psi a continuous bubbling froth existed on the surface. At 1000 psi the froth was found only in the valleys of a closely spaced ($\sim 75 \mu$) ridge-valley surface structure. The fluctuating surface structure is apparently the source for the observed gas phase turbulence. Thus, the schlieren observations of the gas phase during combustion appear to agree with the results obtained from quenched samples.

Impurities in the AP and whether single crystals or pressed polycrystalline wafers were used did not appear to have a significant affect upon the distance between reaction site centers.

B.3 AP-Binder Sandwich Combustion

In the paragraphs that follow, the effects of binder type, binder thickness, AP purity and pressure on the characteristics of the sandwich deflagration will be discussed. When referring to binder thicknesses, 'thin' will be used to describe a thickness of less than 64 microns, 'medium' will be used to describe binder thicknesses of about 100 to 200 microns, and 'thick' will be used for binder thicknesses of approximately 400 microns.

The data obtained from the burning of the SC-UHP AP with CTPB are not discussed since the depth of the sandwich burners used was much larger than the depth of the pressed polycrystalline sandwich burners, allowing much averaging through the flame to occur.

B.3.1 Effect of Binder Composition

Color schlieren taken of PP-UHP AP with thin PBAA and HTPB binders at 300 psig (below the PDL of AP) showed that both were burning with a small laminar flame. A peak temperature existed in the gases above the visible flame and was located above the binder. The peak temperature above the binder was the result of the cool nitrogen atmosphere within the combustion bomb and a single closed (visible) flame above the binder.

When burned at 300 psig, sandwiches made with PP-UHP AP and medium binder thickness of PBAA, HTPB, and PU all exhibited a closed laminar flame with a well-defined peak temperature at the center of the binder (see Figure 12). The combustion of the PBAA sandwich and the HTPB sandwich appeared to be very similar. Both exhibited a relatively large single visible flame. Both visible flames appeared to be laminar, as did the AP deflagration. The PU sandwich burned with a very small flame and the AP deflagration appeared more turbulent than either the PBAA or HTPB sandwiches. This was felt to be due to the binder melt flowing over the AP surface as noted by Boggs and Zurn (8).

Comparison of the color schlieren of sandwiches of PBAA and HTPB with a thin binder thickness, taken at 500 psig (Figure 13) showed that the gases above the sandwich were very close to being turbulent in both cases. No significant difference was evident for the two binders. In both cases the visible flame actually was two flames, one on each side of the binder which protruded above the burner surface. This caused the double blue to red shift seen in these photographs with a temperature minimum above the binder post. The visible flames for the PBAA and the HTPB binders were both approximately the same size.

Comparison of PBAA, HTPB, and PU sandwiches burned at 500 psig with a medium binder thickness showed that all had approximately the same degree of visible flame "turbulence" and flame size. All three exhibited the two flame structure mentioned above for the thin binder case. The PU sandwich surface was almost flat as compared to the slight concave surfaces of the PBAA and HTPB sandwiches. This was once again attributed to the flow of PU binder melt over the AP surface. The visible flame of the PU sandwich appeared more laminar than either the PBAA or HTPB sandwiches, but the gases above the AP appeared slightly turbulent for all three burners.

In summary, sandwiches made with PBAA and HTPB behaved in a very similar fashion with small amounts of binder flow. However, PU appeared to readily flow over the adjacent AP surface, causing the gases evolving from the surface to be more turbulent below the PDL of AP and the surface regression to be flatter above the PDL of AP.

B.3.2 Effect of Binder Thickness

Variation of the binder thickness for sandwiches made with PU were not studied.

Comparison of PBAA sandwiches taken at 300 psig for thin, medium, and thick binder thicknesses indicated that increasing the binder thickness at

this pressure tended to force the deflagration to have a more laminar appearance. Also the visible flame height and width increased as the binder thickness was increased. The more laminar appearance and the larger flames associated with the larger binder thickness appeared to result from an increased binder height with thicker binders. At 500 psig the trends were seen to be the same as for 300 psig, with the exception that the larger binder thickness did not seem to force the deflagration toward a laminar appearance. The visible flame for all binder thicknesses at 500 psig exhibited the two flame structure. The gases above the AP appeared to be somewhat turbulent for the 500 psig cases.

Color schlieren of HTPB sandwiches taken at 300 and 500 psig with thin and medium binder thicknesses indicated the same behavior as for the PBAA sandwiches.

In summary, increasing binder thickness appears to increase the height of the binder protrusion above the AP surface. The increased binder height causes larger visible flames and at pressures below the PDL of AP has a quiescent affect on the gases evolving from the surface.

B.3.3 Effect of Pressure

Comparison of the schlieren for a medium binder thickness of PBAA, with pressures ranging from 100 psig to 600 psig, showed a marked change in the steadiness of the gases evolving from the burner surfaces. To approximately 500 psig there was a discernible single temperature peak in the combustion gases which was located above the binder. Large scale mixing became more and more pronounced as the pressure was increased. As the AP regression rate increased with pressure, sandwich surfaces became flatter.

As pressure was increased from 100 to 400 psig, the flame height increased. From 400 to 500 psig, the flame height decreased and the visible flame transitioned from the single closed structure to the two flame structure.

This resulted from the increased protrusion of the binder above the burner surface. At approximately 500 psig the gases above the AP became turbulent.

For the thin binder thickness the deflagration appeared more turbulent at 300 psig than at 500 psig. This apparent anomaly resulted from the deep, rounded notch burned into the surface of the burner at 300 psig, which caused much large scale mixing of the AP decomposition products. Close investigation showed a larger amount of mixing close to the burner surface at 500 psig, which is above the PDL of AP. Otherwise, the same trends as those discussed above for the medium binder thickness were observed.

Color schlieren of PBAA sandwiches with a thick binder (taken at 300 and 500 psig) showed the same trends as discussed above except that the protruding binder tended to stabilize the visible flame.

HTPB exhibited the same behavior as noted for the PBAA sandwiches except that with the medium binder thickness, flame height decreased with increased pressure.

Color schlieren were also taken of PU sandwiches with medium binder thickness burned at 300 psig and 500 psig. The visible flame size increased noticeably with pressure. The deflagration appeared turbulent at 300 psig possibly due to binder melt flowing over the AP. At 500 psig there was much large scale mixing, and a much more turbulent appearance of the deflagration. The two flame structure was evidenced by the indication of a minimum temperature in the gases directly above the binder.

From the above results, it was concluded that the turbulence level in AP decomposition products increased as pressure was increased from below to above the PDL of AP.

It was also observed that at pressures below the PDL of AP, the visible flame had a single closed structure with one peak temperature in the center above the binder. As the pressure is increased above approximately

350 psi the AP begins to deflagrate. At a pressure somewhere between 400 and 500 psi (depending on binder type, etc.) the AP begins to deflagrate more rapidly than the AP/binder flame, leaving a protruding binder. Once this occurs, the single closed flame is divided into the two flame structure, one on each side of the binder. These flames do not rise vertically above the surface, but rather are canted outward over the deflagrating AP as a result of the binder-protrusion. Thus, even without appreciable binder flow onto the AP, the visible flames become unsteady or "turbulent" as a result of the AP deflagration products interacting with the AP/binder diffusion flame. Binder flow onto the AP further aggravates the situation, causing more visible "turbulence."

One last item deserves mention in this section. In nearly all of the schlieren a distinct yellow-green color was observed to occur periodically. This usually occurred in the gas phase above the burner surface but occasionally it also occurred near the surface of the burner. In the gas phase this yellow-green color was always present with some blue color adjacent to it. Also, it seemed to form in abundance when any irregular portion of the AP protruded well above the region of maximum surface regression. The yellow-green color was observed to occur with all AP crystals, including the single crystal burners. A color film of a PP-UHP AP/HTPB sandwich burned at 500 psig was taken with the schlieren color matrix removed. Therefore, it yielded back-lighted (with the mercury arc) photographs. Light yellow streaks were present in the gas phase. This yellow color with the blue schlieren color may be what caused the yellow-green color observed in the gas phase.

This bright yellow-green color was probably linked to the phase change of AP from an orthorhombic structure to a cubic structure which occurs at approximately 243°C . However, no proof of this conjecture can be offered except the observation of the same color in the single crystal AP just below

the surface. Another possible explanation of the phenomenon is the emission or absorption of light by some product of the AP decomposition.

B.3.4 Effect of AP Purity

Color schlieren were taken of PP-UHP AP/HTPB and PP-COMM AP/HTPB with a medium binder thickness and were burned at 300 psig. The PP-COMM burner surface exhibited the extreme notching characteristic of all burns made with commercial grade AP in this study. The notching effect resulted from the fact that the AP decomposition/deflagration rate for commercial grade AP was less than for UHP-AP. This notching led to much surface generated turbulence for the commercial grade AP.

Color schlieren were also taken of PP-UHP AP/HTPB and PP-COMM AP/HTPB with medium binder thickness and burned at 500 psig. The same observations that were made for the 300 psig case were noted for this case.

It was concluded that impurities in commercial grade AP (notably sulfated ash and tricalcium phosphate) inhibit the deflagration rate. The lower AP deflagration rate causes the sandwich to burn in a notched configuration which increases the turbulence level of the combustion gases.

V. CONCLUSIONS

1. Compaction time is more important than compaction pressure for obtaining high density polycrystalline AP pellets that exhibit single crystal burning rates.
2. Initial crystal size used in pressed polycrystalline AP pellets does not affect burning rate if proper fabrication methods are employed.
3. Additional tests with polycrystalline AP pellets have confirmed the findings reported in Reference 10, namely (a) impurities normally found in commercial grade AP (tricalcium phosphate and sulfated ash) significantly reduce the burning rate and appear to increase the low pressure deflagration limit, (b) the burning rate acceleration sensitivity of AP at 500 and 1000 psi results from surface melt effects, and (c) since AP burning rate decreases with increasing acceleration directed normal and into the burning surface, AP/AP melt interactions with binder/binder melt are the apparent cause of burning rate acceleration sensitivity of nonmetallized composite propellants.
4. Schlieren observations of deflagrating AP agree with the results obtained from quenched samples. The distinct change in the AP surface structure observed by Boggs and Kraeutle (4) when increasing pressure from 800 to 1000 psi (from a continuous bubbling froth to a fine ridge-valley structure with froth in the valleys) was readily apparent in the gas phase with color schlieren photography.

Below 800 psi, distinct reaction sites on the AP are approximately constant in size and are also readily apparent in the color schlieren photography.
5. The turbulence of the gases evolving from the deflagrating surface of AP becomes apparent as pressure is increased. To pressures of approximately 800 psig, the gases very close to the surface appear

laminar. However, the laminar region transitions to turbulent a short distance above the surface. At 1000 psig, the gases are turbulent very near the regressing surface. The fluctuating surface structure (melt, local reaction sites, etc.) is apparently the source of the observed turbulence.

6. The subsurface phase change zone observed in deflagrating AP decreased in thickness with increasing pressure and was not detectible at 1000 psig.
7. AP appears to burn in a pulsating, thermal layer manner.
8. At pressures below that which yields a planar sandwich regression, the visible flame is a closed laminar flame. As pressure is increased the binder protrudes above the regressing surface and splits the flame into two regions, one on each side of the binder. These canted flames interact with the AP deflagration products, causing a visibly "turbulent" flame. Binder flow onto the AP appears to augment the level of turbulence.
9. Sandwiches made with PBAA and HTPB behave in a very similar fashion with small amounts of binder flow. However, PU appears to readily flow over the adjacent AP surface, causing the gases evolving from the surface to be more turbulent below the PDL of AP and the surface regression to be flatter above the PDL of AP.
10. Increasing binder thickness appears to increase the height of the binder protrusion above the AP surface. The increased binder height causes larger visible flames and at pressures below the PDL of AP, has a quiescent affect on the gases evolving from the surface.
11. Impurities in AP retard regression rate and cause AP-binder sandwiches to burn in the notch configuration characteristic of pressures below the PDL of AP. The notch configuration induces considerable turbulence in the gases evolving from the surface.

12. Turbulence level of AP-binder sandwich combustion is a function of pressure, AP purity, surface configuration and the degree of binder flow onto the AP.

In conclusion, high speed color schlieren photography has been shown to be a valuable tool in the study of AP and AP-binder sandwich combustion. Behavior postulated from normal photography and quenched sample examination have been confirmed by examining the temperature profiles in the gas phase during actual combustion.

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TABLE I AP SPECIFICATIONS

<u>Designation</u>	<u>Crystal Size</u>	<u>Principal Impurities (wt%)</u>
UHP (ultra high purity)	38.1 % > 297 μ 81.9 % > 211 μ 99.6 % > 104 μ	Sulfated Ash 0.01%
*Propellant Grade with conditioner (regular 200 μ rounded)	3-11% > 297 μ 13-43% > 211 μ 85-98% > 104 μ	Chlorides 0.07% Sulfated Ash 0.40% Tricalcium Phosphate 0.20%
*Propellant Grade (regular 200 μ rounded)	3-11% > 297 μ 24-52% > 211 μ 85-96% > 104 μ	Na and K 0.03% Ash 0.06% Chlorides 0.05%

*Typical values from manufacturer data sheet

TABLE II

BINDER DATA

<u>PBAA</u>					
Batch No.	PBAA (gm)	EPON 828 (gm)	Cure Time (hr)*	Cure Temp (°C)	
1	3.2041	0.6147	96.0	72	
2	3.2041	0.6147	97.3	72	
3	3.2041	0.6147	96.0	72	
4	3.2401	0.6147	96.0	72	
5	3.2401	0.6147	96.0	72	
6	3.2401	0.6147	96.0	72	
7	3.2401	0.6147	96.0	72	
<u>HTPB</u>					
Batch No.	HTPB (gm)	IPDI (gm)	Cure Time (hr)*	Cure Temp (°C)	
1	3.0000	0.2000	96.0	60	
2	3.0000	0.2000	96.0	60	
3	3.0000	0.2000	168.0	60	
4	3.0000	0.2000	168.0	60	
<u>PU</u>					
Batch No.	Adiprene (gm)	Castor Oil (gm)	1,4 Butane Diol (gm)	Cure Time (hr)*	Cure Temp (°C)
1	7.2000	2.7900	0.0100	120.0	48

*First three hours in vacuum in excess of 28 in. Hg.

TABLE III
TESTS CONDUCTED

Propellant	Binder Type	Binder Thickness (microns)	Pressure (psig)
PP-UHP AP ¹	----	----	300, 320, 330, 340, 345, 348, 350, 355, 358, 360, 380, 400, 450, 500, 800
PP-COMM AP ²	----	----	350, 360, 380, 400, 500
PP-COMM AP ³ w/TCP	----	----	400, 500, 950, 1000
SC-UHP AP ⁴	----	----	200, 400, 500, 800, 1000
PP-UHP AP	PBAA	47 to 439	100, 300, 400, 500, 600
PP-UHP AP	HTPB	37 to 94	300, 500
PP-UHP AP	PU	115	300, 500
PP-COMM AP	HTPB	100	300, 500
PP-COMM AP w/TCP	HTPB	90 to 94	300, 500
SC-UHP AP	CTPB	55 and 192	300, 500

1. Pressed polycrystalline, ultra-high purity AP.
2. Pressed polycrystalline, commercial grade AP.
3. Pressed polycrystalline, commercial grade AP with TCP.
4. Single crystal, ultra-high purity AP.

TABLE IV
EXPERIMENTAL APPARATUS

Schlieren light source	1000 watt mercury vapor arc lamp
External lighting	1200 watt SLM-1200 projector
Color film	Kodak Ektachrome 7241
Light-source chopper location	Just ahead of mercury vapor arc lamp
Knife edge	Blue and red filter matrix
Focusing lens	610 mm, f6
Framing rate	7500 PPS
Camera shutter	1/2.5

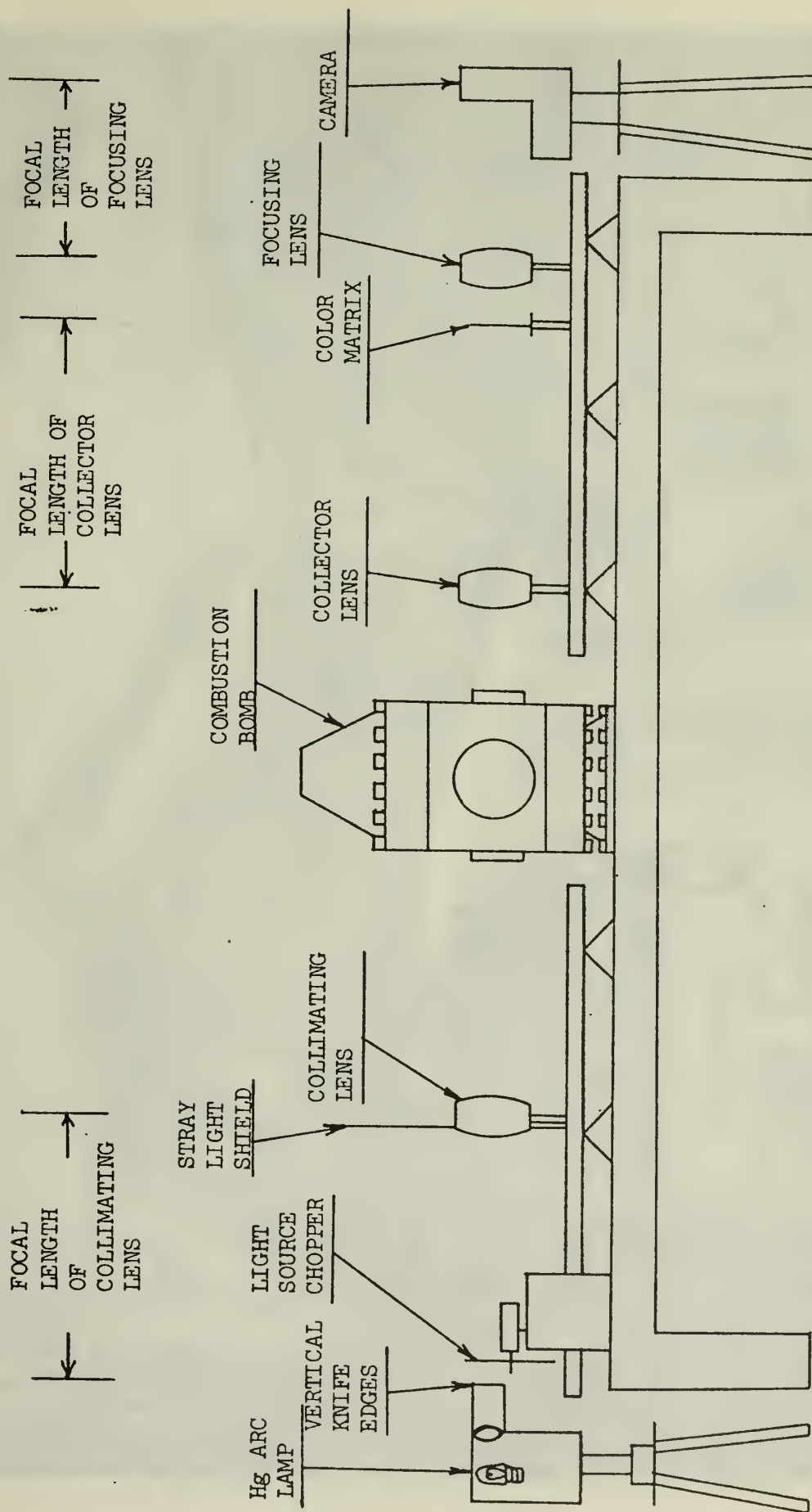


FIGURE 1. SCHEMATIC OF EQUIPMENT ARRANGEMENT

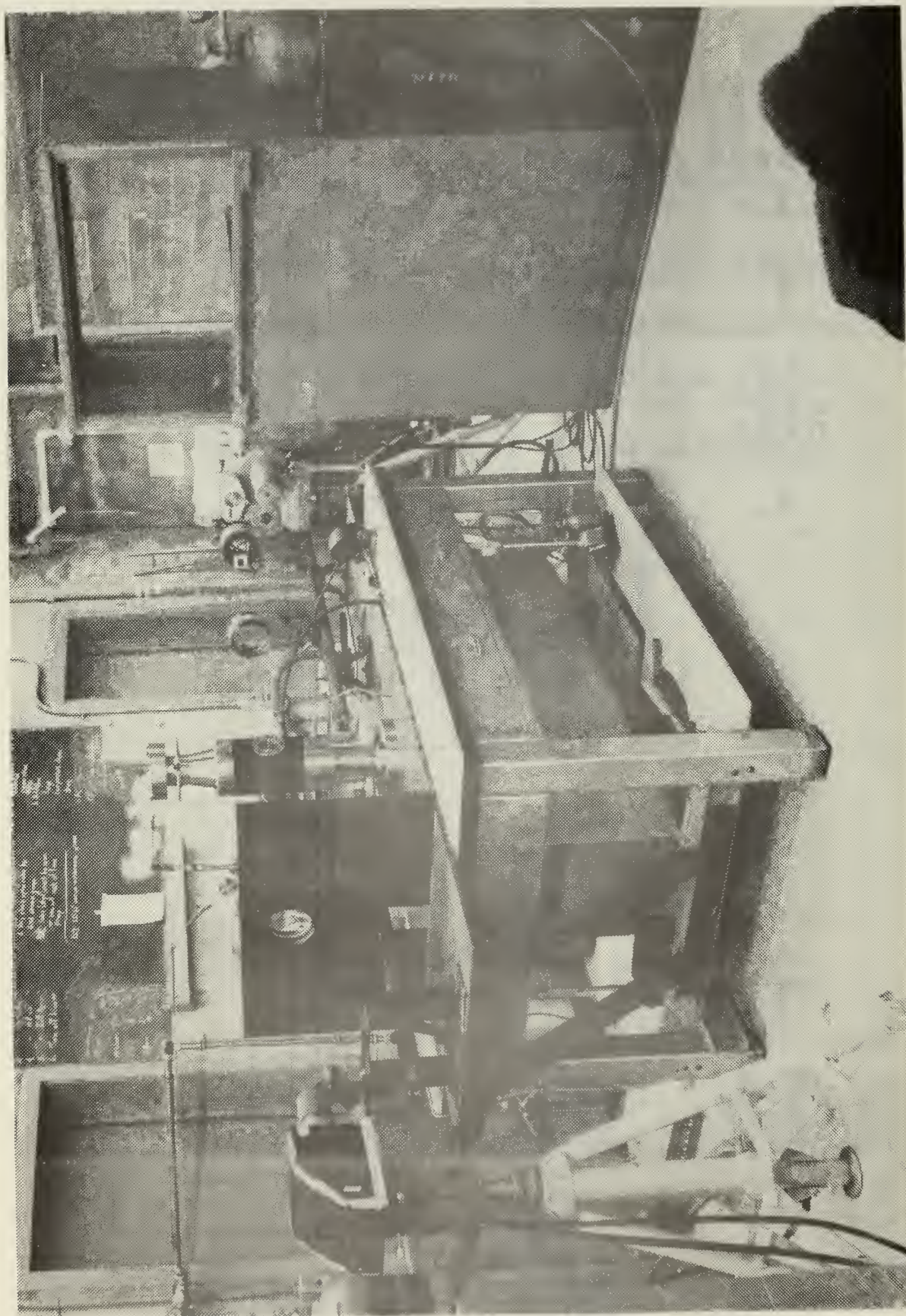


FIGURE 2. PHOTOGRAPH OF APPARATUS

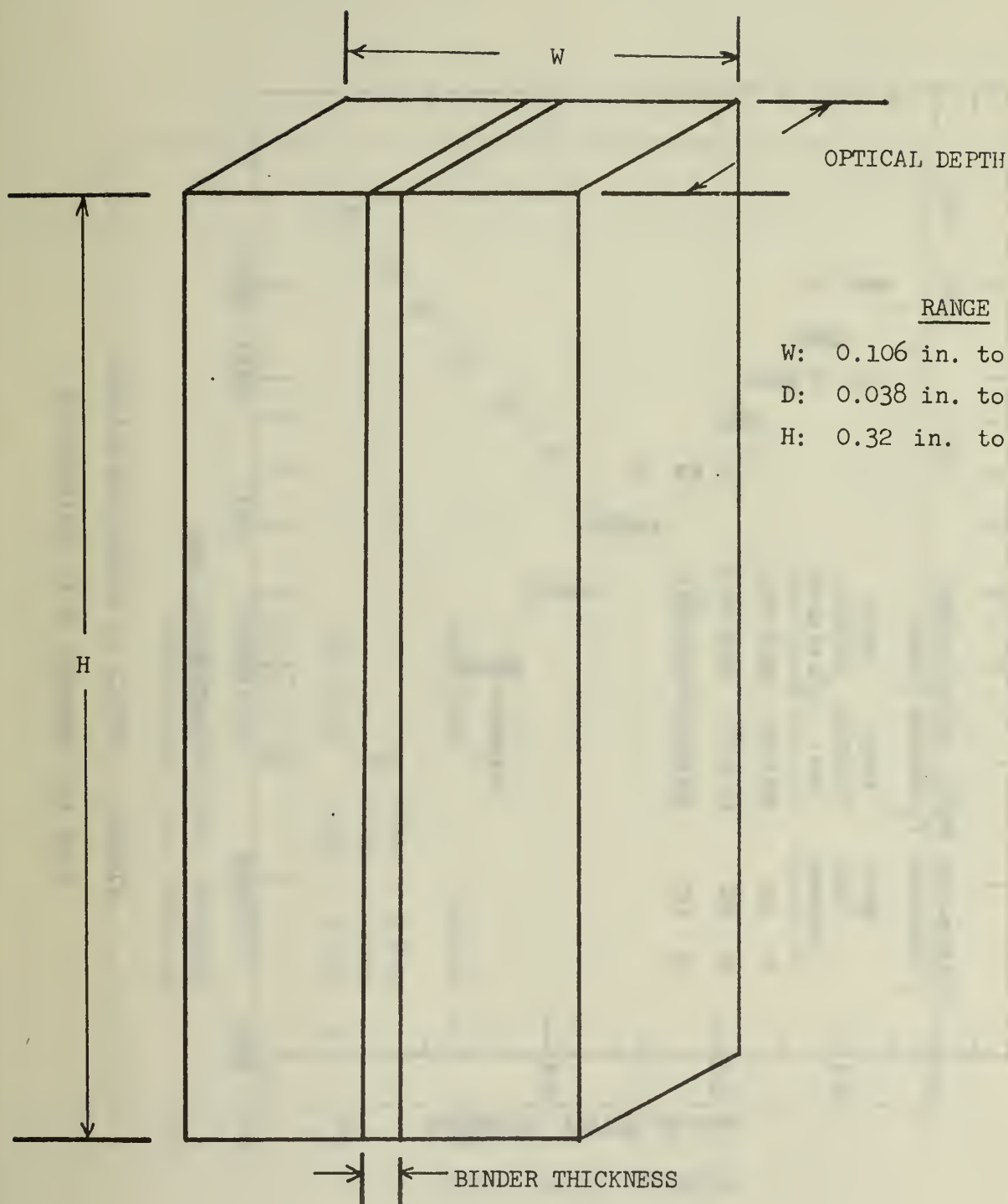


FIGURE 3. SANDWICH BURNER DIMENSIONS

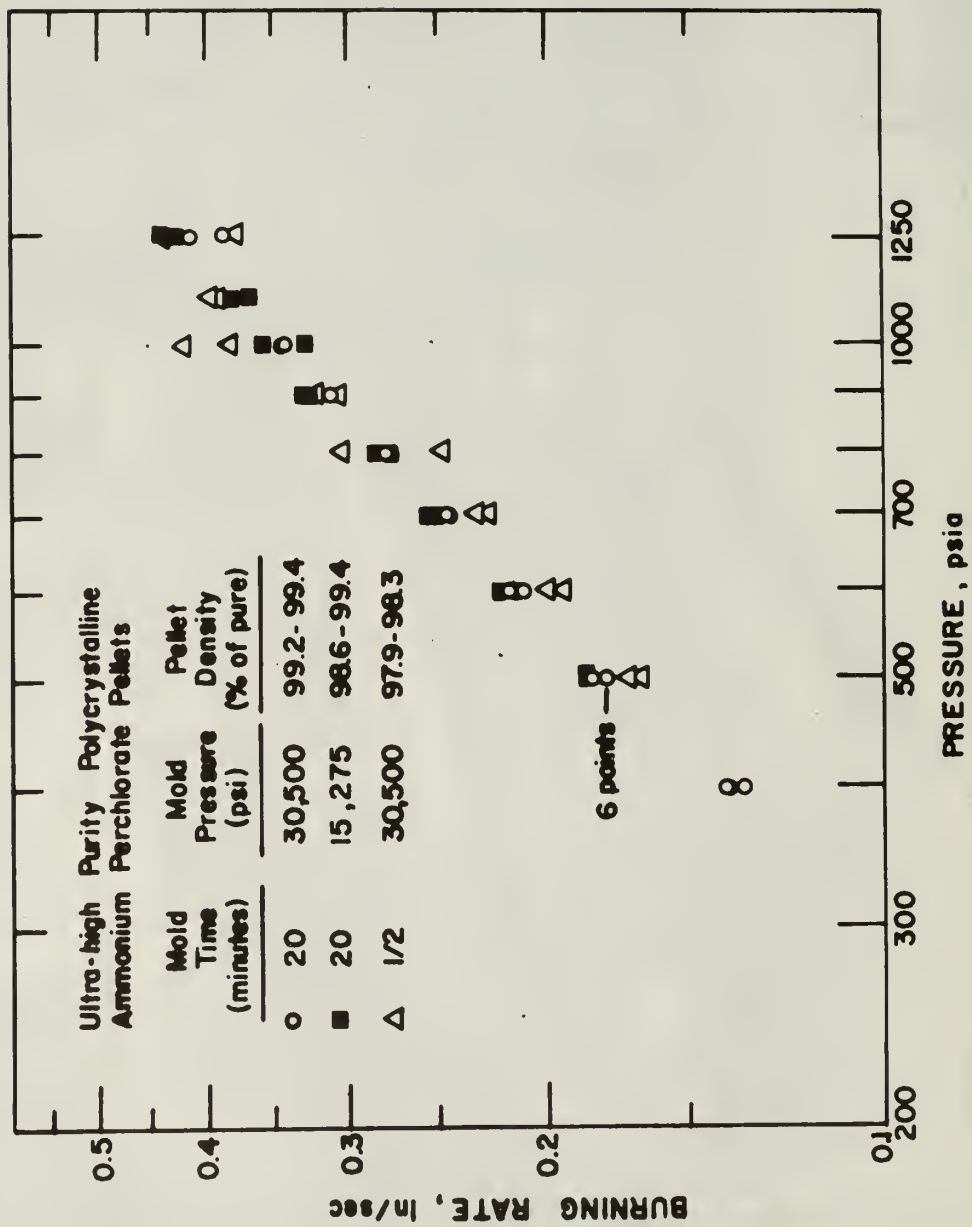


FIGURE 4. EFFECT OF COMPACTION MOLDING
TIME AND PRESSURE ON AP DEFLAGRATION

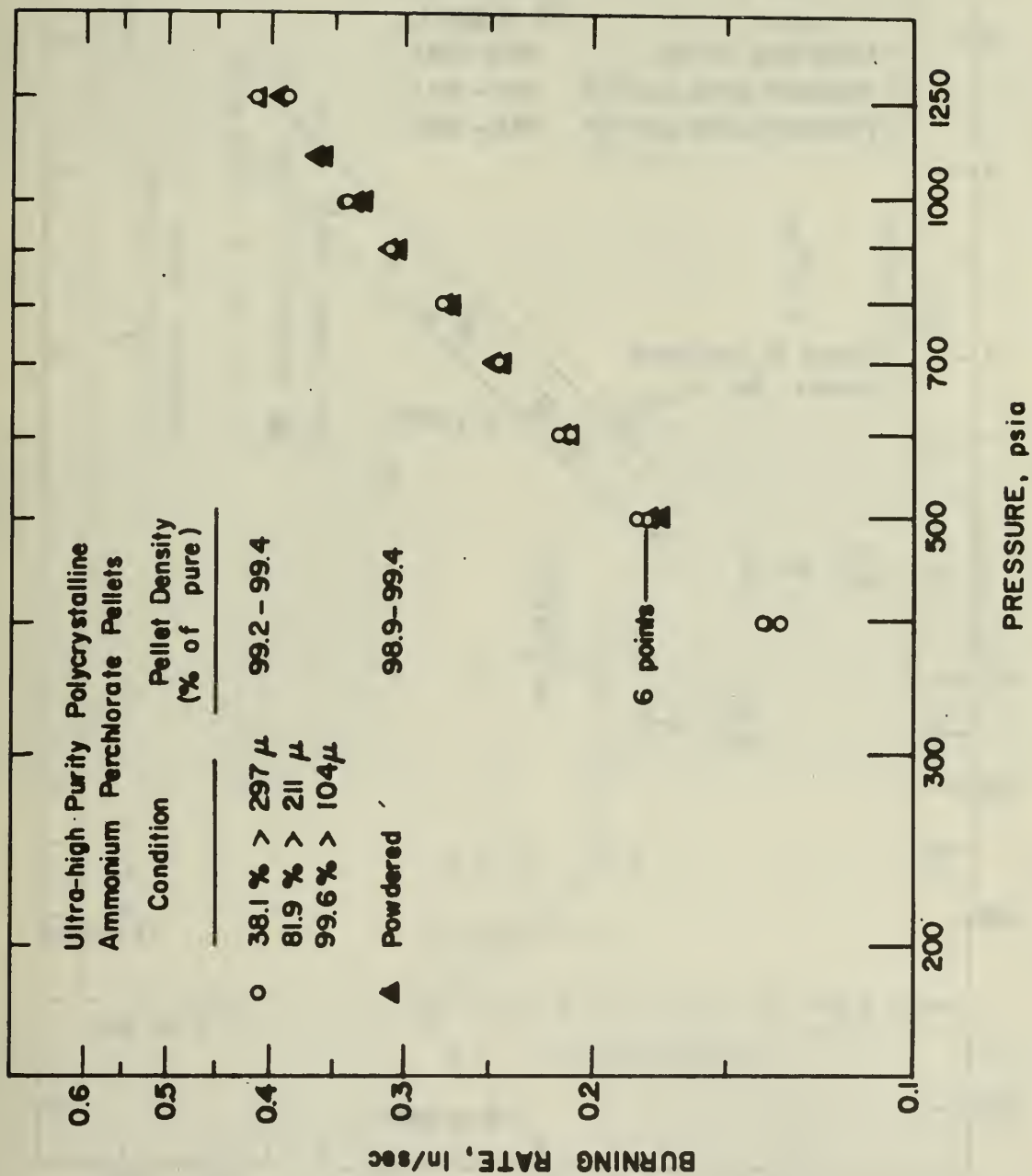


FIGURE 5. EFFECT OF INITIAL AP CRYSTAL SIZE ON PELLET BURNING RATE

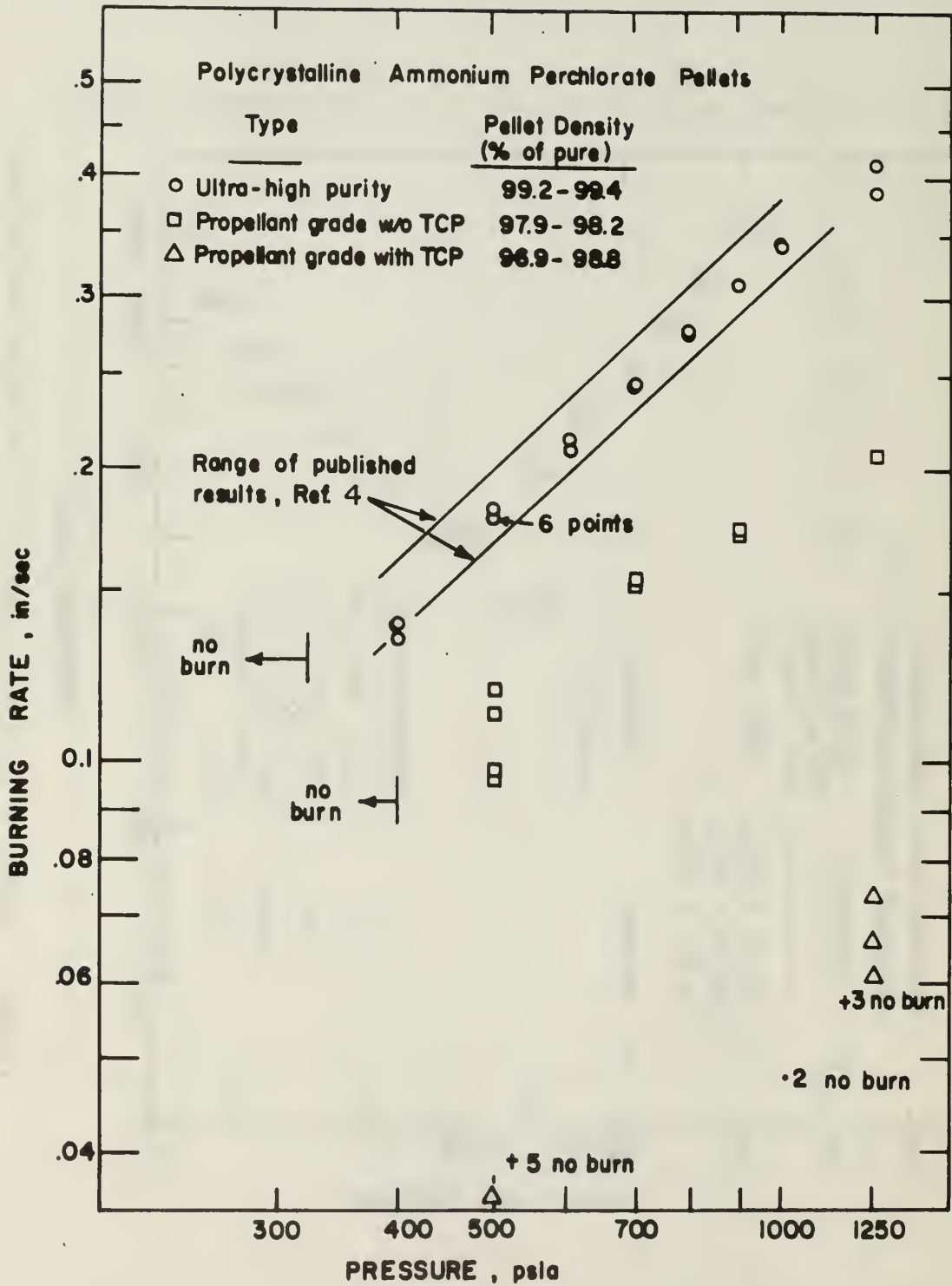


FIGURE 6. EFFECT OF IMPURITIES ON AP BURNING RATE

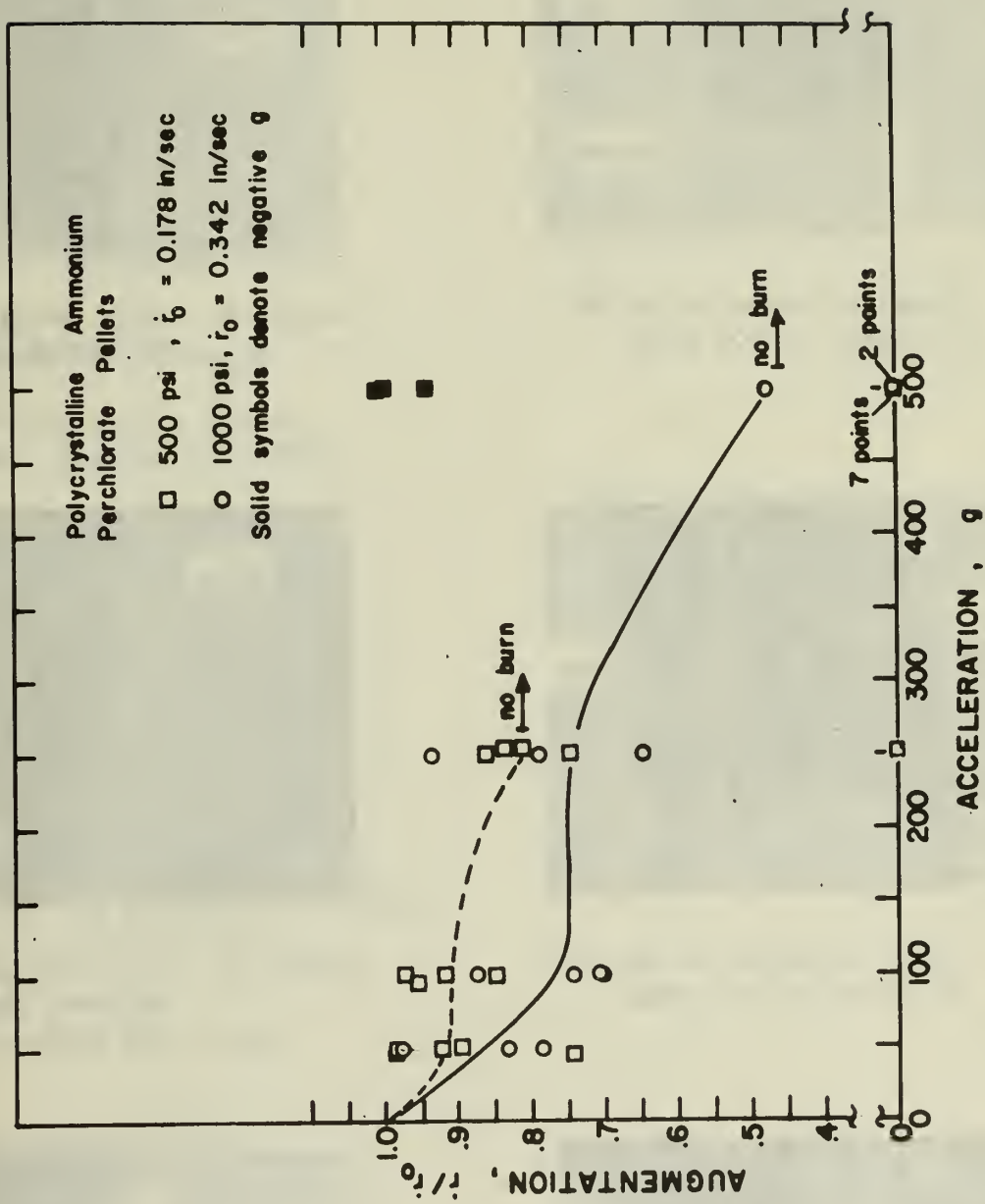


FIGURE 7. EFFECT OF ACCELERATION ON THE BURNING RATE OF AP

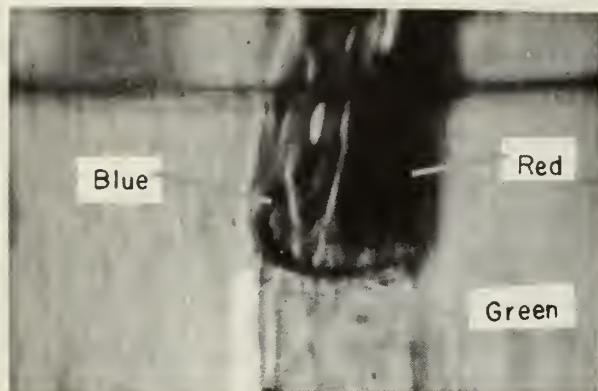


Figure 8. Color Schlieren of SC-UHP
AP Burned at 400 psig

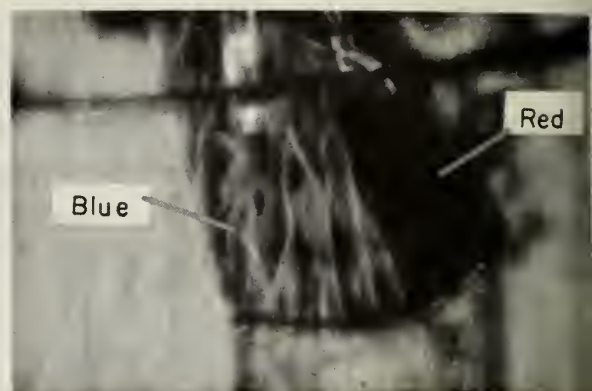


Figure 9. Color Schlieren of SC-UHP
AP Burned at 500 psig

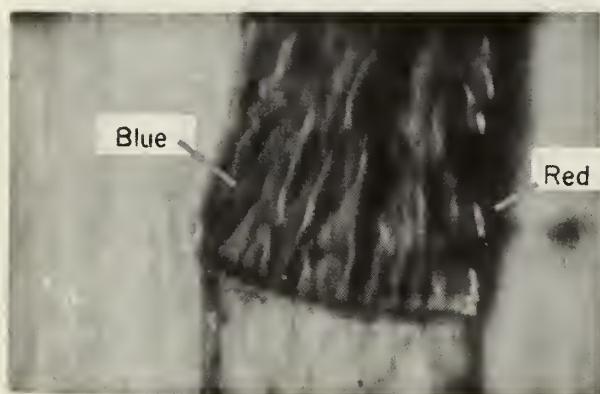


Figure 10. Color Schlieren of SC-UHP
AP Burned at 800 psig

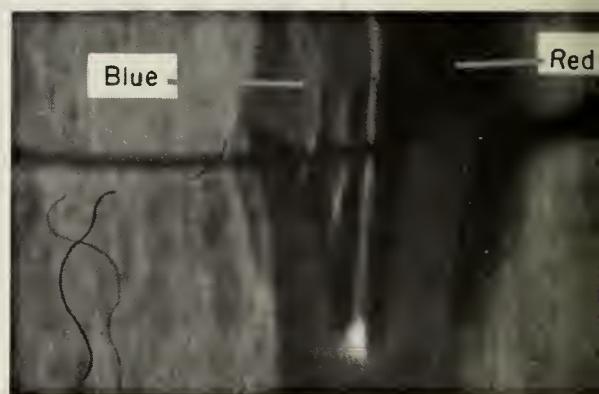


Figure 11. Color Schlieren of PP-UHP
AP/PBAA, 94 micron binder,
100 psig



Figure 12. Color Schlieren of PP-UHP
AP/PBAA, 81 micron binder,
300 psig

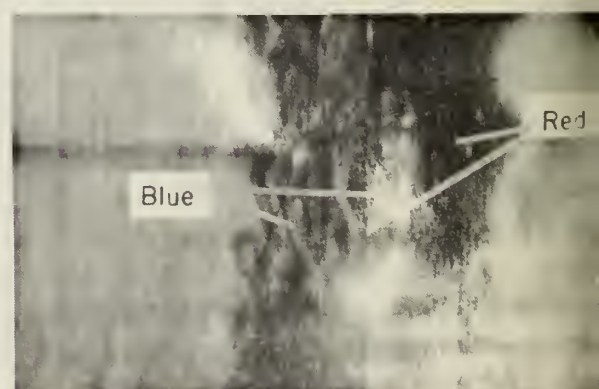


Figure 13. Color Schlieren of PP-UHP
AP/PBAA, 47 micron binder,
500 psig

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11. SUPPLEMENTARY NOTES		12. SPONSORING MILITARY ACTIVITY	
3. ABSTRACT			
<p>Single crystal and pressed polycrystalline ammonium perchlorate (AP) wafers, pressed polycrystalline AP pellets, and ammonium perchlorate-binder (polybutadiene acrylic acid, hydroxyl terminated polybutadiene, polyurethane, and carboxyl terminated polybutadiene) sandwiches were burned in a schlieren equipped combustion bomb and in a centrifuge mounted combustion bomb. High-speed color schlieren and regular motion pictures were taken of the combustion process to determine the effects of pressure, binder type, binder thickness, and purity of ammonium perchlorate on the AP deflagration. AP pellet fabrication techniques and burning rate acceleration sensitivity were also investigated.</p>			

KEY WORDS

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